EXTRACTION OF PALM OIL FROM PALM MESOCARP USING SUB-CRITICAL R134a

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To my beloved family and friends

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ABSTRACT

Experiments involving extraction of palm oil from treated palm fleshy mesocarp were carried out using a dynamic method to determine palm oil yield and solubility in sub-critical 1,1,1,2-tetrafluoroethane (R134a). Effects of crucial process parameters including R134a pressure, temperature and flowrate as well as sample pre-treatment on palm oil yield and solubility were examined. R134a pressure was varied from 60 to 100 bar whereas temperature, from 40^{o} to 80^{o}C. The palm oil extracts were analyzed using gas chromatography (GC) analysis and ultraviolet-visible (UV-Vis) Spectrophotometer to determine the compositions of fatty acids and β-carotene extracted. The experiments were designed using response surface methodology (RSM) to model the extraction yield of palm oil as a function of pressure and temperature. The experimental palm oil solubility data was successfully correlated using Chrastil model, density-based model and temperature-pressure (T-P) model by means of multiple regressions for prediction purposes. The coefficient of determination, R^2, values for the empirical models were 93.68%, 96.38% and 93.97%, respectively. Analysis of variance (ANOVA) showed that palm oil yields and solubility were highly temperature-dependent and moderately pressure-dependent, with p-value of <0.05 at 95% significant level. Sub-critical R134a was a suitable solvent for palm oil extraction since the degree of extraction (defined as the ratio of yield by R134a to Soxhlet) was 93.34%. Substantial oil yields and solubility at much lower pressure than those required by supercritical carbon dioxide proved that sub-critical R134a is an alternative solvent for palm oil extraction even though supercritical carbon dioxide leads to higher oil yields.

ABSTRAK

Pengekstrakan minyak kelapa sawit daripada mesokarpa buah sawit dilakukan secara eksperimen melalui satu kaedah dinamik untuk menentukan keterlarutan minyak sawit dan kebolehan pengekstrakan di dalam subgenting (1,1,1,2-tetrafluoroethane) R134a dengan mengambil kira kesan parameter proses termasuk tekanan, suhu, kadar alir R314a dan rawatan sampel. Tekanan bagi R134a diubah dari 60 ke 100 bar manakala suhu adalah dari 40^{o} ke 80^{o}C. Ekstrak minyak sawit yang dianalisis melalui kromatografi gas dan Spektrofotometer UL-Nampak untuk menentukan komposisi asid lemak dan β-carotina yang diekstrak. Eksperimen telah direkabentuk melalui kaedah permukaan sambutan (RSM) untuk permodelan hasil pengekstrakan minyak sawit terhadap suhu dan tekanan sistem. Data keterlarutan minyak sawit yang diperolehi telah berjaya berkolerasi menggunakan model Chastil, model berasaskan ketumpatan dan model suhu-tekanan (T-P) melalui kaedah regresi berganda. Nilai pekali penentuan, R^2, bagi setiap model empirikal tersebut adalah 93.68%, 96.38% and 93.97%. Analisis varians (ANOVA) menunjukkan keterlarutan dan hasil ekstrak minyak sawit adalah sangat dipengaruhi oleh suhu dan separa dipengaruhi oleh tekanan apabila p-value<0.05 pada 95% tahap kepentingan bagi kedua-dua faktor suhu dan tekanan. Keputusan eksperimen menunjukkan pelarut R134a boleh diaplikasikan proses pengekstrakan minyak sawit memandangkan darjah pengekstrakannya terhadap pengekstrakan Soxhlet mencapai sehingga 93.34%. Keterlarutan dan hasil pengekstrakan yang tinggi walau pada tekanan rendah membuktikan pelarut R134a boleh diaplikasikan dalam pengekstrakan minyak sawit. Bagaimanapun, pelarut karbon dioksida lampau genting memberikan kadar ekstrasi yang lebih tinggi daripada pelarut subgenting R134a.

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LIST OF ABBREVIATIONS / SYMBOLS / NOTATIONS

Abbreviations

AAD	-	Average absolute deviation
AAPD	-	Average absolute percentage deviation
CF	-	Cooked-flaked
CFC	-	Chlorofluorocarbons
CO_2	-	Carbon dioxide
CPO	-	Crude palm oil
C12:0	-	Lauric acid
C14:0	-	Myristic acid
C16:0	-	Palmitic acid
C16:1	-	Palmitoleic acid
C18:0	-	Stearic acid
C18:1	-	Oleic acid
C18:2	-	Linoleic acid
C18:3	-	Linolenic acid
C20:0	-	Arachidic acid
DG	-	Diacylglycerols
EPO	-	Evening Primrose oil
FAME	-	Fatty acid methyl esters
FDA	-	Food and Drug Administration
FFA	-	Free fatty acids
FFB	-	Fresh fruit bunch
GC	-	Gas chromatography
GCMS	-	Gas chromatography mass spectrophotometer
GRAS	-	Generally Recognised as Safe
HC	-	Hydrocarbons

HCFC	-	Hydroclorofluorocarbon
HFCs	-	Hydroflourocarbons
HPLC	-	High-pressure liquid chromatography
MDI	-	Metered dose inhaler
MG	-	Monoglycerides
N_2O	-	Dinitrogen Monoxide
OF	-	Objective function
P_c	-	Critical pressure
P_r	-	Reduced pressure
PCB	-	Polychlorobiphenyls
POO	-	Palmitoyl-dioleoyl-glycerol
POP	-	Dipalmitoyl-oleoyl-glycerol
PPO	-	Dipalmitoyl-oleoyl-glycerol
SCF	-	Supercritical fluid
SEM	-	Scanning Electron Microscope
SFE	-	Supercritical fluid extraction
SF_6	-	Sulphur Hexafluoride
TIC	-	Total Ion Chromatogram
TG	-	Triglycerides
UC	-	Uncooked-chopped
UF	-	Uncooked-flaked
VOC	-	Volatile organic compound

Symbols

α	-	Alpha
β	-	Beta
μ	-	Dipole moment
ε	-	Dielectric constant
π^*	-	Polarity/polarizability parameter
a_1	-	Constant depends on total heat of reaction
a_2	-	Constant depends on molecular weights of solute and fluid
c	-	Solubility
k_1	-	Slope of the solubility isotherm

ΔH	-	Heat of reaction
ρ_f	-	Density of fluid
Y	-	Total oil yields in sub-critical as g oil/g sample
a_{ij}	-	Coefficient of the function
P	-	Absolute pressure
P_C	-	Critical pressure
P_r	-	Reduced pressure ≡ P / P_C
T	-	Absolute temperature in Kelvin
T_C	-	Critical temperature
T_r	-	Reduced temperature ≡ T / T_C
S	-	Palm oil solubility (g oil/kg R134a used)
R^2	-	Regression coefficient
ρ	-	R134a density

Notations

Subscripts

calc	-	Denotes calculated value
exp	-	Denotes experimental value
f	-	Denoted fluid

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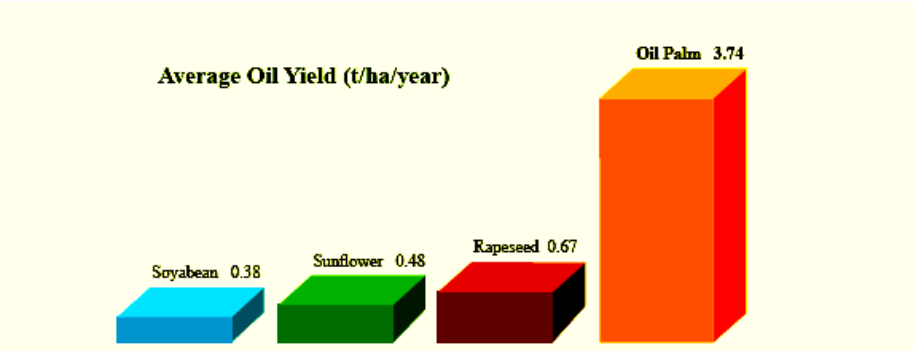
CHAPTER 1

INTRODUCTION

1.1 Outlook of Palm Oil Industry

Palm oil is an important source of food and a major source of lipid. Steady increase in the world population increases the demand for palm oil as an important source of edible oils and fats. Almost 90% of the world palm oil production is traded as edible oils and fats. Palm oil also account for about 13% of the total world production of oils and fats, and is expected to overtake soybean oil as the most important vegetable oil (Sundram *et al.,* 2003). This phenomenon arises due to the unique characteristics of palm oil, particularly its potential health benefits. Therefore, high yields of the oil palm throughout the year are essential to meeting the high global expert market demand.

The oil palm produces the highest tonnage of oil per hectare per year with minimum use of land see Figure 1.1. The Figure 1.1 shows a comparison of oil palm with other major oil crops in the world. The average yield of oil palm produced in a year was 3.74 tonne/hectare/year and contributed to 31.11% of the world's total oil crop production. This followed by soybean oil (29.78%), rapeseed oil (15.50%) and sunflower oil (9.37%) (MPOC and MPOB, 2007).



Oil Crop	Oil Production (mil tonnes)	% of Total Oil Production	Average Oil Yield (t/ha/year)	Planted Area (mil ha)	% Total Planted Area
Soyabean	35.26	29.78	0.38	92.63	42.27
Sunflower	11.10	9.37	0.48	22.95	10.47
Rapeseed	18.36	15.50	0.67	27.29	12.45
Oil Palm (mesocarp)	36.84	31.11	3.74	9.86	4.50
Total*	118.42			219.15	

Figure 1.1: Comparison of palm oil with other oil crops for year 2006 (MPOC and MPOB, 2007)

Currently, most of the world's production of palm oil comes from South-East Asia, in particular Malaysia and Indonesia (Sundram *et al.,* 2003). Malaysia and Indonesia continues to be the largest contributors, with the respective average of 51% and 36% production of palm oil between the periods of 2002-2006 as shown in Figure 1.2 (MPOC and MPOB, 2007). Therefore, as the biggest producer and exporter of palm oil and palm oil products, Malaysia plays an important role in fulfilling the growing global needs for oils and fats.

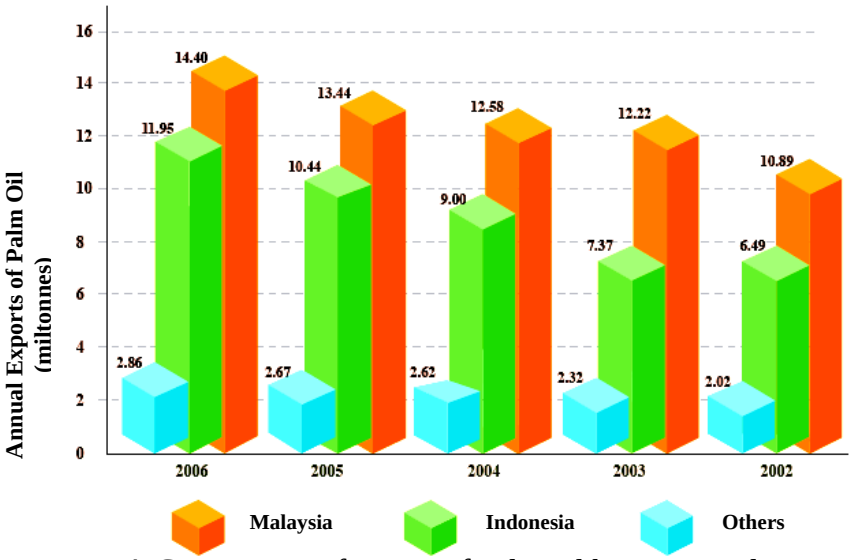


Figure 1.2: A Comparison of export of palm oil between Malaysia and other producer (MPOC and MPOB, 2007).

1.2 Palm Oil Extraction

Palm oil is produced from the fruit of oil palm tree (*Elaeis* Guineensis) which originated in West Guinea. The average weight of each bunch varies between 10-30kg and individual fruits are generally in the range of 8-20g. As shown in Figure 1.3, the individual fruit are made up of an outer skin (the exocarp), a pulp (mesocarp) containing the palm oil in a fibrous matrix; a central nut consisting of a shell (endocarp); and the kernel (the seed), which itself contains an oil, quite different from palm oil, resembling coconut oil (Sundram *et al.,* 2003).

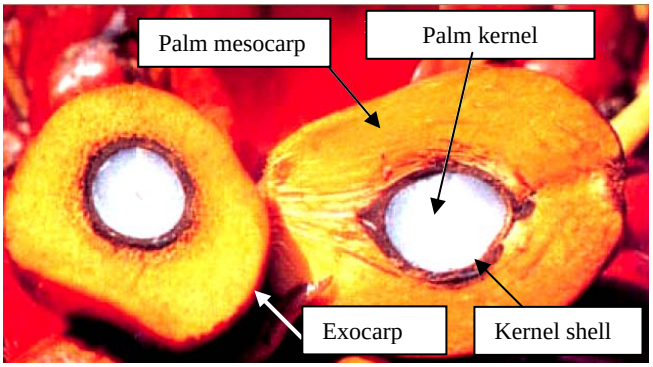


Figure 1.3: Anatomy of oil palm fruit
(MPOC and MPOB, 2007)

A unique feature of the oil palm is that it produces two types of oil - palm oil is extracted from mesocarp, and palm kernel oil is extracted from the palm kernel. Palm oil is rich in carotenoids, (pigments found in plants and animals) from which it derives its deep red colour, and the major component of its triglycerides is the palmitic acid which is a saturated fatty acid; hence it is a viscous semi-solid, under ambient typical condition and becoming solid fat in temperate climates.

1.2.1 Conventional Processing

The common technique of extracting palm oil from fruits in Malaysian palm oil milling is by mechanical pressing using hydraulic press or screw press. In the pre-processing stage, fruits bunches are collected from the plantation and delivered to the mill as soon as possible to avoid the increase of undesired content i.e. free fatty acid (FFA) due the enzymatic reaction. The oil extraction process involves several steps namely sterilization, bunch stripping or threshing, digestion of fruits, followed by extraction of oil and clarification and purification as shown in Figure 1.4.

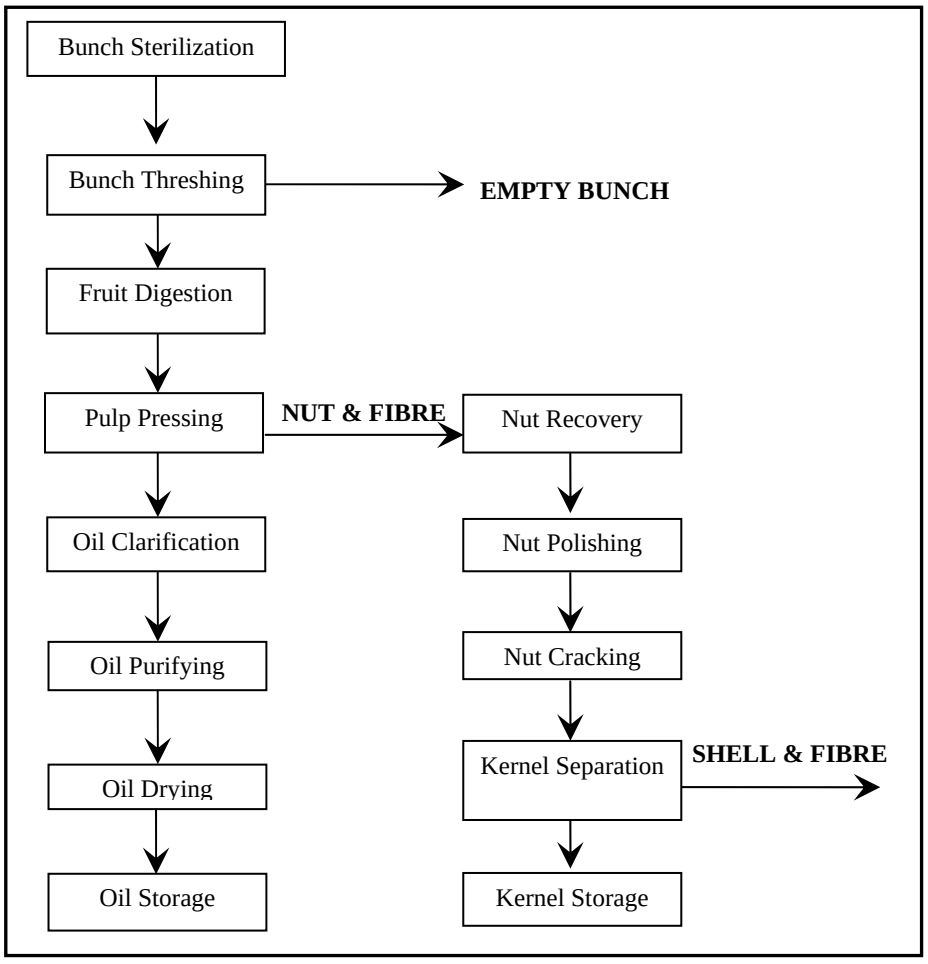


Figure 1.4: Flow diagram of conventional palm oil extraction process.

Fresh fruit bunches are steam sterilized at around 140^{o}C for a period of 75 to 90 minutes. The sterilizing or steaming of the fruits are to facilitate separation and threshing of bunches to free the palm fruit. However, the main objectives are to deactivate hydrolytic enzymes responsible for the breakdown of oil to free fatty acid, to coagulate and facilitate the breaking of oil cells. In the bunch threshing step, the fruits are stripped and separated from fruit bunches in a rotary drum. Fruits are knocked out of the bunch as they passed through the stripper. The detached fruits are collected and conveyed into the digester (Mustapa, 2004).

Digestion involves the meshing up of the fruits under steam-heated conditions and pressing out the crude palm oil. Generally, twin-screw press is used to press out the oil from the digested mesh. The crude oil is further purified and dried for storage. The solid wastes from the milling operations are empty fruit bunches, palm fibre, and palm kernel shell. The residue from the press consists of a mixture of fibre and palm nuts which were then processed to produce palm kernel. Normally, the extraction rates of fresh fruit bunch are dependent on the size and weight of fruits. Fruits weighing more than 20 kilograms would typically an extraction rate of between 19–21%, whereas fruits weighing is less than 10 kilograms, typically will have oil extraction rate of about 15–16% (Mustapa, 2004).

The best quality crude palm oil quality must have FFA percentage of less than 5% whereas the moisture content is less than 0.1% and the percentage of dirt is not more than 0.01%. The highly rated commercial oil palm fruit is type Tenera, a hybrid of Durra and Pisifera (Baryeh, 2001 and Sundram *et al.,* 2003). Tenera has a shell of less than 3mm thick 60-96 % mesocarp, 3-20% shell and 3-15% kernel making it tenable to high extraction yield.

1.2.2 Carotenoids in Palm Oil

One of the uniqueness of palm oil is, its high content of carotenoids. Typical crude palm oil contains 500-700ppm of carotenes. Together with tocopherols, carotenoids contribute to the stability and nutritional value of palm oil. Therefore, the extraction and recovery of the carotenes would give a significant added value to the oil. Unfortunely, these valuable carotenes are destroyed and removed during conventional palm oil refining to give a light coloured oils as required consumers, rendering them unavailable for recovery and use (Ooi *et al.,* 1996 and Gast *et al.,* 2001).

Besides, the conventional processing of palm oil also leaves high content of carotenoids in pressed palm fibers. The pressed palm fibers found are a good source of carotene. The residual palm fibers from palm oil production contain between 4000 and 6000 ppm of carotenoids, six times higher than that found in crude palm oil (Franca and Meireless, 1997). Until now, the residue is a waste product which is typically burned in palm oil mills, regardless of the high carotenoid contents. (Birtigh *et al.,* 1995 and Franca and Meireless, 1997).

A number of methods have been developed to recover and extract carotenoids from crude palm oil including solvent extraction, adsorption, precipitation and transesterification-distillation. However, to-date, only transesterification-distillation and transesterification-solvent extraction have been scaled-up to industrial practise. These methods are energy-intensive processes since the esters must be vacuum-distilled and the carotenes may undergo thermal degradation. As an alternative SFE technology was introduced more than 20 years ago to take advantage of the lower operating temperature preventing degradation of carotene during extraction. The SFE method has wide attention due its advantages over traditional processing technology.

1.3 Supercritical Fluid Extraction (SFE) Technology

Supercritical fluid extraction (SFE) is a powerful technique in a separation process which uses a supercritical fluid (SCF) as a solvent. The behaviour of supercritical fluids observation was first observed and reported by Baron Cagniard de la Tour in 1822. From his early experiments, the critical point of a substance was first discovered. However, the first workers to demonstrate the solvating power of supercritical fluids for solids were Hannay and Hogarth in 1879. They found that increasing the pressure caused the solutes to dissolve and that decreasing the pressure caused the dissolved materials to precipitate as a “snow”. This behaviour is fundamental to the understanding of the supercritical fluids extraction (SFE) technology (Taylor, 1996).

A pure component is said to be in supercritical state when its temperature and pressure are higher than the critical values (*Tc* and *Pc*, respectively). The fluid above the T_c and P_c cannot be liquefied regardless of the applied pressure. It has desirable properties that make it suitable for challenging an extraction process i.e. changes with a slight variation in pressure and temperatures near the critical point. The fluid exhibits high density similar to liquids, and low viscosity similar to gases. The high densities of SCFs contribute to greater solubilization of compounds while low viscosity enables penetration in solids and allows flow with less friction. On the other hand, surface tension and heat of vaporization is relatively very low for SCFs (De Castro, 1994).

1.4 Advantages of SFE technology

The applications of SFE technology have developed as a faster and less solvent-intensive alternative to traditional extraction schemes as a result of the critical fluids having more promising properties over conventional solvent. Supercritical fluid extraction (SFE) methods involves a few steps, thereby leading to short analysis time and reduced sample transfer, and hence smaller analytical errors. In addition, by using SFE technology, the recovery of analytes is precisely and reliability compared to conventional method i.e. Soxhlet extraction as isolation of the analytes is completes. This is due to the solvent power, polarity and temperature of SCF that play role as primary factor to a success extraction process. The crude oils obtained from SFE method are easily be refined compared to conventional method since the SFE extracts contain fewer impurities (De Castro, 1994). Throughout the SFE process technology, the raw materials and extracts are not subjected to elevated temperatures, thereby minimising the risk of product thermal degradation. In addition, by supercritical fluid extraction technology the operating condition can be operated under a wide range to extract selectively the any active compound or to produce with improved functional or nutritional characteristics.

SFE technology is cleaner and less hazardous than conventional extraction since 90% of the technology applications developed so far use CO_2 as a solvent, which is non-toxic and inflammable properties. Unlike liquid solvents (n-hexane, ether, methylene chloride), supercritical CO_2 poses no fire risk and leaves no environmentally hazardous wastes. The properties of gas-like diffusivity and viscosity, zero surface tension, and liquid-like density combined with the pressure-dependent solvent power of a supercritical fluid have provided the stimulation for applying supercritical fluid (SCF) technology to a range of separations problems experienced in many segments of industry (Taylor, 1996).

1.5 Problem Background

Currently, SFE applications have been very limited, and are focused on the use of CO_2 as a supercritical solvent as alternative to conventional separation, i.e. Soxhlet extraction. In fact, supercritical CO_2 has been accepted as a common and standard supercritical solvent and is the most popular and typically employed as a solvent for most supercritical extraction applications. Even though SFE technology has been established and known during the past 30 years, however, commercialization of the technology is still limited (Rizvi *et al.,* 1986). There are only a few industrial SFE applications that have been reported such as for decaffeination of coffee and tea and extraction of hops for brewing (Rizvi *et al.,* 1986 and Ooi *et al.,* 1996).

This limitation may be attributed to the high capital investment associated with plant start-up and intense operation due the higher pressure of up to 500 bar required by using carbon dioxide as a solvent to allow satisfactory extraction or fractionation process (Rizvi *et al.,* 1986). The high-pressure operation can contribute to the high capital cost and operating cost to maintain the high pressure. This is one of the factors that have contributed to the limited the commercialisation of SFE process using CO_2.

Some attempts have been made to use carbon dioxide at low pressure ranging from 40-100 bar. However, it was found that the solubility of the compound of interest in CO_2 is rather low (Maxwell, 1996, Catchpole and Von Kamp, 1997 and Catchpole *et al.,* 1998). Previously, Stahl *et al.* (1980) had studied the extraction of seed oils by liquid CO_2, found that the inferior mass transport properties and solute fluxes caused the approach for oilseed processing inadequate. In addition, King and Bott (1993) has noted that there is no economic incentive in the near-critical CO_2 process.

The discovery of a new or alternative low-pressure solvent having the same advantages as that of CO_2 is therefore necessary in order to capitalise on the superiority of SFE technology over traditional technique. Subcritical R134a is

suggested as a low-pressure alternative (Catchpole and Proells, 2001 and Wood *et al.,* 2002) to supercritical CO_2 since it has been found to have comparable solvent properties to CO_2 (Lagalante *et al.,* 1998) in addition to being able to extract analytes at low temperature and pressure (Hansen *et al.,* 2000 and Simões and Catchpole, 2001).

In addition, numerous SFE applications have focused on the extraction of vegetables and seeds such as soybean (Lee *et al.,* 1991), canola (Fattori *et al.,* 1988 and Temelli, 1992), Buriti (Franca *et al.,* 1999), sunflower (Krimiti *et al.,* 2001 and Salgın *et al.,* 2005), and palm kernel (Hassan *et al.,* 2000) using SC-CO_2 to recover valuable minor components such as tocopherols and *β*-carotene. The extraction of palm oil using SFE technology is envisioned to have a great outlook (Bharat, 2003). However, extraction of palm oil from its fruits using SC-CO_2 has been found to be relatively rare as compared to the application of SFE on other vegetable oils (Bisunadan, 1993 and Lau *et al.,* 2006). Until today, there has been no reported study on the use of R134a as an alternative to CO_2 solvent for the supercritical extraction and recovery of palm oil and its valuable minor components.

1. 6 Problem Statement

Until today, the commercialization of SFE technology has been rather limited. High operation cost associated with high operating pressure needed by CO_2 as a solvent is one of the key factors that limit the commercialisation of SFE technology (Rizvi *et al.,* 1986, Catchpole and Proells, 2001, and Simões and Catchpole, 2002). It is envisioned that the discovery of an alternative solvent that allows operations at significantly lower pressure relative to those required using CO_2 solvent can overcome the practical limitations towards commercialisation. For the solvent to be viable, it should enable the process to achieve comparable or better performance in terms of maximising oil yields while maintaining the quality and stability of the oil through elimination of undesirable compounds. In achieving

economical and cleaner process, it is envisioned that R134a has a promising potential to replace CO_2 for the extraction of crude palm oil based on SFE technology.

1.7 Research Objectives

The aims of this research are:

a) To investigate the capability of R134a as an alternative to CO_2 solvent by determining yields and solubility of palm oil in sub-critical R134a solvent at various pressure and temperature using SFE technology.

b) To investigate the potential of R134a to recover the valuable minor components by measuring the concentration of extracted β-carotene.

1.8 Scope of Work

In order to explore the viability of using R134a as a new solvent, it is essential to establish fundamental data such as solubility. Palm oil extraction from its oil palm fleshy mesocarp was performed to investigate the capability of sub-critical R134a as alternative low-pressure solvent to CO_2 by determining the yields and solubility of palm oil in sub-critical R134a. The potential of R134a solvent to recover the valuable minor component was also investigated by measuring the concentration of β-carotene extracted. The tasks to be accomplished to achieve the objectives of this study include:

- Experiments to determine palm oil solubility in sub-critical R134a at selected range of conditions.

- Investigation of the effects of process parameter (temperature, pressure, flowrate) and effects of sample pre-treatment on the yields and solubility of palm oil and analysis of composition of fatty acids and concentration of β-carotene using Gas Chromatography (GC) and UV-Vis Spectrophotometer respectively.
- Correlation of the solubility behavior with a density-based model and T-P model.
- Prediction of Response Surface Model for extraction yield including effects of pressure and temperature.

1.9 Research Contributions

Three key contributions that have emerged from this work are:

- Equilibrium solubility data for palm oil in sub-critical R134a. To-date, there has been no reported findings on the solubility of palm oil in R134a. Therefore, the solubility data presented in this thesis is a pioneering work in the development of solubility database in SFE field.
- An empirical model on the solubility behaviour of sub-critical R134a provides a significant impetus for future SFE measurement and predictions studies.
- A detailed experimental procedure on the use of sub-critical R134a as an alternative solvent to supercritical CO_2. The experiments conducted in this work will be a breakthrough step to chart the path towards the development of an economical alternative new process for the crude palm oil production and for the recovery of valuable minor components (such as carotenes and tocopherols) in palm oil based on the principle of SFE principles.

1.10 Thesis Outline

This thesis is organised in 6 chapters. Chapter 1 begins with the introduction of the research project i.e. preface on SFE technology related to vegetables oils and fats application. The chapter also include the problem background which had motivated this research, the research objectives and scope of work and the key contributions of this work.

Chapter 2 describes the fundamental theory as well as the physical and chemical properties of palm oil and its minor-components. This chapter also presents an overview of the thermodynamic properties of R134a as well as the most common SFE solvents. These properties are useful for solvent selection. The fundamentals of SFE process and sub-critical conditions are also presented in the chapter. This includes specifications on the parameters influence an extraction process and solubility.

Chapter 3 provides a critical review of the previous works on solubility, SFE applications to vegetables and seeds oils by means of CO_2 with R134a as a solvent. Theory and literature on Response Surface Design for statistical model and mathematical modeling on the solubility behaviour are also included in this chapter

Chapter 4 presents the experimental work procedure for the extraction process. The procedure begins with selection of operating conditions, sample pre-treatment and extraction methodology. It also describes the procedure for analysis of extracted oil sample for compounds such as fatty acids and carotene using Gas Chromatography (GC) and UV-Vis Spectrophotometer, respectively.

The results discussed in Chapter 5 are divided into two parts. The experimental study results are discussed in the first part. These include discussions on the effects of various process parameters on the oil extraction yield as well as solubility; and discussions on the effects of process parameters on the compositions of fatty acids and carotene. In the second part, empirical modeling that describe the

oil yields and solubility characteristics as well as the mathematical models on solubility behaviour based on solvent density are discussed and proposed.

Finally, Chapter 6 presents a summary of the study and the recommendations for future work. Some suggestions are made to guide and improve future experimental work in terms of the use of sub-critical R134a as a solvent.